IS 7168 (1973): Warfarin Sodium Salt Water Soluble Powders
[FAD 1: Pesticides and Pesticides Residue Analysis]

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IS:7168-1973
(Reaffirmed 2012)

Indian Standard

SPECIFICATION FOR WARFARIN SODIUM SALT WATER SOLUBLE POWDERS

UDC 632·958·31 WAR

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

Price Rs 5·00 Gr 3

April 1974

Indian Standard

SPECIFICATION FOR WARFARIN SODIUM SALT WATER SOLUBLE POWDERS

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Indian Standard

SPECIFICATION FOR WARFARIN SODIUM SALT WATER SOLUBLE POWDERS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 18 December 1973, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Warfarin sodium salt water soluble powders are used as baits for the control of rodent pests in homes, fields and warehouses.

0.3 This standard is one of a series of Indian Standards on pest control products. Other standards in this series are given on page 12.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test **for** warfarin sodium salt water soluble powders.

2. REQUIREMENTS

2.1 Description-The material shall consist of a homogeneous mixture of warfarin sodium, technical; sodium benzoate; tetrasodium ethylene diamine tetra-acetate; and sugar. It shall be in the form of uniformly blended free-flowing powder and free from extraneous matter which may limit its suitability for baiting rodents or impair its effectiveness.

2.1.1 Warfarin, sodium, technical used in the formulations of the powders shall conform to IS :5551-1970†.

*Rules for rounding off numerical values (*revised*).
†Specification for warfarin sodium, technical.

2.2 **The material shall comply with the requirements given in Table 1.**

TABLE 1 REQUIREMENTS FOR WARFARIN SODIUM SALT -WATER-SOLUBLE POWDERS

SL No.	CHARACTERISTIC	REQUIREMENT	METHODS OF TEST	
			Ref to Appendix	Ref to Clause of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Warfarin sodium (technical), percent by mass	Nominal value as declared on the container with the appropriate tolerance of percent content of the nominal value (see 2.2.1)	A	—
ii)	Tetrasodium ethylene diamine tetra-acetate, percent by mass	3·0 to 3·3	B	—
iii)	Sodium benzoate, percent by mass	3·0 to 3·3	C	—
iv)	Matter insoluble in water, percent by mass, *Max*	0·1	D	—
v)	Moisture, percent by mass, *Max*	1·0	E	—
vi)	Sieving requirement material passing through 150-micron IS Sieve†, percent by mass, *Min*	95	—	12.1
vii)	Sucrose. content	To make up the rest of the mass	F	—

*Methods of test for pesticides and their formulations.

†BS Sieve 100 and 150, ASTM test Sieve 100 and 140, and Tyler test Sieve 100 and 150 have apertures within limits specified for 150-micron IS Sieve.

2.2.1 **The appropriate tolerances to be applied shall depend** upon the declared percent **nominal value of warfarin sodium, technical** (*see* Table 1) **and they shall be as given below:**

Nominal Value, Percent	*Tolerance Limits, Percent*
up to 10	$^{+10}_{-5}$ **of the nominal value**
Above 10 and up to *50*	± *5* **of the nominal value**
Above *50*	$^{+5}_{-3}$ **of the nominal value**

3. PACKING AND MARKING

3.1 The material shall be packed in clean and dry airtight containers made of galvanized steel sheet, tinplate, mild steel glass or plastic as agreed between the purchaser and the manufacturer.

3.2 The containers shall be securely closed, sealed airtight after filling them with the material and shall bear legibly and indelibly the following information in addition to the provisions as given under the rules of the Insecticides Act:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Net mass of the contents;

f) The word ' POISON' in distinct, bold capital letters in red; and

g) The minimum cautionary notice worded as under:

'KEEP THE MATERIAL AND THE BAITS CONTAINING THE MATERIAL AWAY FROM CHILDREN, DOMESTIC ANIMALS, FOODSTUFFS AND EMPTY FOODSTUFF CONTAINERS. DO NOT USE THESE CONTAINERS FOR STORAGE OF FOOD STUFFS. WEAR RUBBER GLOVES TO DISPENSE THE BAITS. IN CASE OF POISONING CALL A PHYSICIAN. SPECIFIC ANTIDOTE FOR WARFARIN POISONING IS VITAMIN K_1. BLOOD TRANSFUSION MAY BE NECESSARY.'

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — **The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.**

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in Indian Standard method of sampling of pesticides and their formulations (*under preparation*).

NOTE - **Until the standard under preparation is published, the matter shall be subject to agreement between the concerned parties.**

5. TESTS

5.1 Tests shall be carried out as prescribed in the appropriate appendices given in col and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (see IS :1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* **1,** *Item* **(i)**]

DETERMINATION OF WARFARIN SODIUM SALT CONTENT

A-l. APPARATUS

A-l.1 Spectrophotometer — Beckmann model DU, or equivalent instrument with 1 cm quartz cells.

A-2. REAGENT

A-2.1 **Reference Solution** — Weigh accurately 1·29 g of tetrasodium ethylene diamine tetra-acetate, 1·29 g of sodium benzoate, and 40·3 g of sucrose. Transfer to a 1-litre volumetric flask and make up to the mark with distilled water. Dilute a 5-ml aliquot to 100 ml with 0·1 N sodium hydroxide solution so that the *p*H of the final solution lies between 10 and 11.

A-3. PROCEDURE

A-3.1 Weigh accurately about 43 g of the sample into a 1-litre volumetric flask, dissolve in distilled water, and make up to the mark. Dilute a 5 ml aliquot to 100 ml with sodium hydroxide solution so that the *p*H of the final solution lies between 10 and 11. Pipette a sufficient amount (about 3 ml) of the final solution into a l-cm quartz cell and with the spectrophotometer set at a maximum sensitivity determine its absorbance at 308 nm against the reference solution.

*Specification for water, distilled quality (***revised).***

A-4. CALCULATION

A-4.1 Warfarin sodium salt content, percent by mass

$$= \frac{E \times 330{\cdot}3 \times 100 \times 1000 \times 100}{1{\cdot}42 \times 10\,000 \times 1000 \times 5 \times m}$$

$$= \frac{E \times 46{\cdot}52}{m}$$

where

E = absorbance of the final solution at 308 nm, and

m = mass in g of the material taken for test.

APPENDIX B

[*Table* 1, Item (ii)]

DETERMINATION OF TETRASODIUM ETHYLENE DIAMINE TETRA-ACETATE CONTENT

B-l. APPARATUS

B-l.1 Spectrophotometer — Beckmann model DU, or equivalent instrument with 1 cm quartz cells.

B-2. REAGENTS

B-2.1 Standard Phosphate Buffer Solution — Disodium hydrogen phosphate (0·2 M), the *p*H of which has been adjusted to 11·0 with sodium hydroxide solution.

B-2.2 **Standard Stock Solution-Dissolve** 4·0 g of tetrasodium ethylene diamine tetra-acetate in distilled water and make up to 700 ml.

B-2.2.1 *Standard Working Diluted Solution -Transfer* 0·25-, 0·50-, 0·75-, 1·0-, 1·5-, 2·0-, and 3·0-ml aliquots of the standard stock solution to seven 100-ml volumetric flasks and dilute to 95 ml in each flask with the phosphate buffer solution. Titrate with 0·25 percent copper sulphate solution to the first permanent turbidity and add 2 drops in excess. Make up to 100 ml with the phosphate buffer solution and centrifuge until clear.

B-3. PROCEDURE

B-3.1 Preparation of Standard Curve — Determine the absorbance of the standard working dilutions at 340 nm by means of the spectrophotometer, using the phosphate buffer solution as reference. Plot the observed absorbances against percentage contents (m/v) of tetra sodium ethylene diamine tetra acetate in dilutions.

B-3.2 Analysis of Sample-Dissolve 4·50 g of the sample in distilled water and make up to 100 ml. Transfer a 10-ml aliquot to a 100-ml volumetric flask and add 85 ml of the phosphate buffer solution, titrate with 0·25 percent copper sulphate solution to the first permanent turbidity, and add 2 drops in excess. Make up to 100 ml with the phosphate buffer solution and centrifuge until clear. Determine the absorbance of the final solution at 340 nm by means of the spectrophotometer, using the phosphate buffer solution as reference. Calculate the percentage content of tetrasodium ethylene diamine tetra-acetate by comparison with the standard curve.

APPENDIX C

[*Table* **1,** *Item* (iii)]

DETERMINATION OF SODIUM BENZOATE CONTENT

C·1. PROCEDURE

C-1.1 Weigh accurately about 2 g of the sample into a 500-ml flask for titration. Add 75 ml of ether, 20 ml of distilled water, and 5 drops (or more if necessary) of methyl orange indicator and titrate with 0·5 N hydrochloric acid, mixing intimately the water and ether layers by vigorous stirring or shaking, until a permanent orange colour is produced in the aqueous layer. Carry out a blank determination on a solution containing 0·06 g of sodium salt of warfarin, 0·60 g of tetrasodium ethylene diamine tetra-acetate, and 18·7 g sucrose in 75 ml of ether and 20 ml of distilled water.

C-2. CALCULATION

C-2.1 Sodium benzoate content, percent by weight $= \frac{(a-b) \times 7{\cdot}025}{m}$

where

a = volume in ml of 0·5 N hydrochloric acid used for the sample,

b = volume in ml of 0·5 N hydrochloric acid used for the blank, and

m-massing.

APPENDIX D

[*Table* 1, *Item* (iv)]

DETERMINATION OF MATTER INSOLUBLE IN WATER

D-1. PROCEDURE

D-1.1 Weigh accurately about 5 g of the sample into a 250-ml beaker, add 50 ml of distilled water and heat to 30°C, while stirring. Filter the solution through a dry, tared Gooch crucible and wash twice with 100 ml portions of distilled water at approximately 30°C. Dry to constant mass in an oven at 1 10°C, cool and weigh.

APPENDIX E

[*Table* 1, *Item* (*v*)]

DETERMINATION OF MOISTURE CONTENT

E-1. PROCEDURE

E-1.1 Weigh accurately about 2 g of the sample into a. dry tared dish (nickel, platinum or aluminium) and dry for 2 hours under atmospheric pressure at 100 to 102°C, then cool in a desiccator and weigh. Dry again for 1 hour, or until the change in mass is not greater than 2 mg. Report the loss in mass as moisture content.

APPENDIX F

[*Table* 1, *Item* (iiv)]

SUCROSE CONTENT

F-1. APPARATUS

F-1.1 Polarimeter -graduated in one-tenths of a degree.

F-2. PROCEDURE

F-2.1 Weigh accurately about 28 g of the sample into a 100 ml volumetric flask, dissolve in distilled water, and make up to the mark at 25°C. Determine the angular rotation of this solution at 20°C by means of the polarimeter.

F-3. CALCULATION

F-3.1 Sucrose content, percent by weight $= \frac{151{\cdot}5\,a}{b\,m}$

where

a = angular rotation of the solution at 25°C,

b = length of the polarimeter tube (dm), and

m = mass in g.

(*Continued from page 2*)

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INDIAN STANDARDS

ON

PESTICIDES (TECHNICAL GRADE)

IS:

560-1969 BHC, technical and refined (***second revision)***
563-1973 DDT, technical (***second revision)***
634-1965 Ethylene dichloride carbon tetrachloride mixture (3 : 1 *v/v*) (***revised***)
882-1956 ***gamma-13HC (*** lindane)
1051-1957 Pyrethrum extracts
1052-1962 Dieldrin, technical (***revised)***
1055-1957 Nicotine sulphate solution
1251-1973 Zinc phosphide, technical (*first revision*)
1306-1958 Aldrin, technical
1309-1958 Endrin, technical
1311-1956 Ethylene dibromide (first revision)
1312-1967 Methyl bromide (*first revision*)
1486-1969 Copper oxvchloride, technical (first revision)
1488-1969 2, 4-D sodium, technical (*first* revision)
1682-1973 Cuprous oxide, technical (fungicidal grade) (first ***revision***)
1827-1961 Liquid amine salts of 2, 4-D
1832-1961 Malathion, technical
1833-1961 Diazinon, technical
2125-1962 Phenyl mercury salicylate, technical
2126-1973 Phenyl mercury acetate, technical (*first* revision)
2127-1962 Stabilized methoxy ethyl mercury chloride concentrate
2128-1973 Parathion ethyl, technical (*first* revision)
2353-1963 Phenyl mercury chloride, technical
2354-1963 Ethyl mercury chloride, technical
2355-1963 Stabilized ethoxy ethyl mercury chloride concentrate
2570-1963 Methyl parathion, technical
2863-1964 Chlordane, technical
3898-1966 Zineb, technical
3900-I 966 Ziram, technical
3902-1966 Dimethoate, technical
3904-1966 Thiometon concentrates
4320-1967 Thiram, technical
4321-1967 2,4-D technical
4344-1967 Endosulfan, technical
4345-1967 Binapacryl, technical
4451-1967 Toxaphene, technical
4929.1968 Dichlorvos, technical
4958-1968 Phosphamidon, technical
5278-l 969 Dicofol, technical
5280-1960 Fenitrothion, technical
5526-1939 Coumafuryl, technical
5549-1970 Warfarin bait concentrates
5551-1970 Warfarin, sodium, technical
5552-1970 Warfarin, technical
6432-1972 Heptachlor, technical

AMENDMENT NO. 1 SEPTEMBER 1976

TO

IS : 7168-1973 SPECIFICATION FOR WARFARIN SODIUM SALT WATER SOLUBLE POWDERS

Alteration

(*Page* **4, clause 2.2.1**) **-Substitute** the following for the existing clause:

'2.2.1 ***Warfarin Sodium, Content*** — When determined by the method prescribed in Appendix A, the observed warfarin sodium, content, percent (by mass) of any of the samples shall not differ from the declared nominal value by more than the tolerance limits indicated below:

Nominal -Value Percent	*Tolerance Limit*	
Up to 9	+ 10 − 5	percent of the nominal value
10 and below 50	± 5	
50 and above	+ 5 − 3	

2.2.1.1 The actual value of warfarin sodium, content shall be calculated to the second decimal place and then rounded off to the first decimal **place** before applying the tolerances as stipulated in 2.2.1.'

(AFCDC 6) Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 MAY 1994
TO
IS 7168 : 1973 SPECIFICATION FOR WARFARIN SODIUM SALT WATER SOLUBLE POWDERS

(*Page* 5, clause 4.1) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause 2.2.1 of the standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India